THE EFFECT OF WASTE COOKING OIL METHANOL MOLAR RATIO ON YIELD AND PROPERTIES OF BIODIESEL PRODUCED IN A MINI PLANT

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DECLARATION

I hereby declare that this thesis entitled "T***he effect of waste cooking oil methanol molar ratio on yield and properties of biodiesel produced in a mini plant"*** **is** the result of my own research except as cited in the references. This thesis has not been accepted for any degree and is not currently submitted in candidature of another degree.

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Abstract

Azhar food factory used a lot of cooking oil at least 144 tonne per year for frying and the company produce waste cooking oil about 54 tonne per year and WCO need to dispose in a suitable way without harming the environment. One of the alternative methods proposed is by converting the WCO into biodiesel. A mini plant to produce biodiesel from waste cooking oil was been design, fabricated and installed at the factory. Biodiesel from WCO are difficult and costly when free fatty acid content more is than 2%. There are some problems of WCO such that it can lead to pollution and health risk. Therefore, this research were carried out to propose a simple small pilot plant to produce biodiesel using WCO and to investigate the operation of the plant with WCO as input with various molar ratio which is 6:1, 9:1 and 12:1 respectively with the same parameter such as holding time of 2 hours, temperature of 60^0C and 600 rpm agitation speed. The properties of biodiesel such as Free Fatty Acid (FFA), water content, flash point, acid value, density, kinematic viscosity, and moisture content was investigated to fulfil standard ASTM D7461 and EN14013 Fatty Acid Methyl Ester to determine yield of biodiesel using GC-FID. The FAME value of molar ratio 6:1 is 97.8% meanwhile molar ratio 9:1 value is 97.2 % and molar ratio 12:1 is 95.7% has been achieved. Evaluation and comparison between five percent biodiesel from the plants and B5 petroleum diesel with standards ASTM D975. In this process were made molar the ratio of alcohol 12:1 cost very high is RM421.60, meanwhile molar ratio 9:1 cost RM329.60 and 6:1 molar ratio cost RM246.96. The best yield of methyl ester from waste cooking oil is 6:1 From the result above it can be concluded that the produced WCO-Biodiesel has achieved the standards because it fulfilled ASTM D7461 and EN 14131 standard.

Abstrak

Kilang Azhar food menggunakan minyak masak 144 tan setiap tahun untuk menggoreng dan menghasilkan minyak masak terbuang 54 tan setiap tahun. Minyak masak terbuang perlu dihapuskan dengan kaedah yang bersesuaian tanpa mencemarkan alam sekitar. Salah satu kaedah ialah dengan mengitar semula minyak masak terbuang kepada biodiesel. Loji mini untuk menghasilkan biodiesel dari minyak masak terbuang telah direka, fabrikasi dan dipasang di kilang. Penghasilan biodiesel dari minyak masak terbuang memerlukan kos yang tinggi kerana mempunyai lemak bebas lebih dua peratus . Minyak masa terbuang juga menyebabkan masalah kesihatan dan punca pencemaran. Oleh itu, kajian ini mencadangkan satu loji mini untuk menghasilkan biodiesel daripada minyak masak terbuang dan menyiasat operasi loji dengan pelbagai nisbah molar seperti 6:1, 9:1 dan 12:1 dengan mengunakan parameter tetap seperti suhu 60^0C, 2 jam dan kelajuan putaran 600 rpm. Paiwaian ASTM D7461 digunakan untuk menyiasat sifat-sifat biodiesel seperti lemak bebas, kandungan air, takat kilat, nilai asid, ketumpatan, kelikatan dan kandungan kelembapan dan piawaian EN14214 untuk menentukan peratus hasil biodiesel dengan mengunakan GC-FID. Nilai peratus biodiesel dengan mengunakan nisbah molar 6:1 sebnayak 97.8% manakala 9:1 ialah 97.2% dan 12:1 sebanyak 95.7% . Piawaian ASTM D975 digunakan untuk membuat perbandingan dan pernilaian diantara biodiesel daripada loji sebanyak 5 peratus dengan petroleum diese B5. Dalam penghasilan biodiesel nisbah molar 12:1 lebih mahal dengan jumlah RM 421.60 jika dibandingankan dengan nisbah molar 9:1 sebanyak RM 329.60 dan RM 2463.96 untuk nisbah 6:1. Untuk proses menghasilan biodiesel dengan mengunakan minyak masak terbuang nisbah molar 6:1 adalah nilai peratus yang baik. Kesimpulannya minyak masak terbuang ni berjaya untuk menghasilkan biodiesel dengan memenuhi piawaian ASTM D7467 dan EN14131.

TABLE OF CONTENTS

LIST OF TABLE

LIST OF FIGURE

LIST OF SYMBOL AND ABBEREVIATIONS

AMMC	-	Advanced Manufacturing and Material Centre
ASTM	-	American Society of Testing and Materials
B5	-	Biodiesel 5 %
B10	-	Biodiesel 10 %
B100	-	Biodiesel
C	-	Degree Celsius
CARB	-	California Air Resources Board
CO	-	Carbon monoxide
CO_2	-	Carbon Dioxide
CPO	-	Crude palm oil
EU	-	European Union
EPA	-	Environmental Protection Agency
FAME	-	Fatty Acid Methyl Ester
FFA	-	Free Fatty Acid
FID	-	Flame Ionization Detector
g/cm^3	-	Gram / cubic centimeter
g/mL	-	Gram per milliliter
g/mol	-	Molar mass per gram
GC	-	Gas chromatography
H_2	-	Hydrogen
HC	-	Hydrocarbon
HCI	-	Hydrochloric acid
H_2O	-	Water
H_2SO_4	-	Sulpuric Acid
K	-	Kelvin
kg/h	-	Kilogram/hour
KOH	-	Potassium Hydroxide
$KOCH_3$	-	Potassium methoxide
kpa	-	Kilopascal

MeOH	-	Methanol
MJ/kg	-	Megajoule / kilogram
mm^2/sec	-	Millimeter square /second
MPOB	-	Malaysian Palm Oil Board
MS	-	Mass spectrometry
$NaOCH_3$	-	Sodium methoxide
NaOH	-	Sodium hydroxide
NOx	-	Nitrogen oxides
ppm	-	Parts Per Million
PPO	-	Pure Plant Oil
RBD	-	Refined Bleached Deodorized
RPM	-	Revolutions per minute
SVO	-	Straight Vegetable Oil
TG	-	Triglycerides
UM	-	Universiti Malaya
USM	-	UniversitiSains Malaysia
UTHM	-	University Tun Hussein Onn Malaysia
Vol%	-	Percentage volume
wt. %	-	Weight Percentage
WCO	-	Waste Cooking Oil

LIST OF PUBLICATIONS

Nor Hazwani Binti Abdullah and Sulaiman Haji Hasan, “Identification of methyl ester content from Waste Cooking Oil Using Gas Chromatographic Method” Applied Mechanics and Materials, Vol. 660 (2014) pp 297-300

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Azhar food factory started off as a food marketing company, mainly being part of the small industry in 1991. Its main focus was on the business selling for the surrounding areas in Renggit, Johor. In 1999, Azhar food factory own a processing plant located in Renggit Johor. In June 2011, Azhar food penetrated into the domestic market and our products have been distributed all over Malaysia and Taiwan. Azhar food factory is selling a variety of crisps such as cracker, tapioca chips and snacks all of which are produced by its own factory starting from the raw material, frying, and packing up until marketing. The company business' coverage is focused on convenient stores, wholesalers and retailers.

Table 1.1: Cooking oil and Waste Cooking Oil per year

Cooking Oil		**Waste Cooking oil**	
Time period	**Quantity (tonne)**	**Time period**	**Quantity (tonne)**
1 week	3	1 week	1
1 month	12	1 month	4.5
6 month	72	6 month	27
1 year	144	1 year	54

Azhar food factory used a lot of cooking oil 144 tonne for fraying and produce waste cooking oil 54 tonne per year and WCO need to dispose in a suitable way without harming the environment. One of the specify methods proposed is by converting the WCO into biodiesel. The company has agreed to allow UTHM to assist them in this venture. A mini plant to produce biodiesel from waste cooking oil (WCO) has been design, fabricated and installed at the factory site.

In this study, waste cooking oil from Azhar food factory was processed to biodiesel were free fatty acid more than 2% take from storage 3. Converting into biodiesel need two-step catalyst process and fatty acid methyl ester (FAME) content should be greater than 90% follow by standards EN14103. Biodiesel properties standard musttofulfill the American Standard for Testing and Material (ASTM D7461) and European Union (EN14214). Biodiesel from mini plant will be used for Azhar food factory such as burner, forklift and diesel engine for 5% biodiesel.

1.2 Problem statement

From daily fraying process, a large amount waste cooking oil are produced and factory take alternative to converting to biodiesel. By processing, biodiesel as output the factory can reuse for burner, forklift and diesel engine. This research will evaluate the biodiesel produced by the fabricated plant and provide suggestion to improve it.

A large amount of waste cooking oil (WCO) may effect to pollution increment and health risk. Therefore, to overcome this problem one of the alternative is to convert WCO into biodiesel using a mini pilot plant.

Biodiesel from WCO are difficult and costly were free fatty acid contain more than 2%. Most food factory in Renggit Johor are small factories and cannot afford the high cost of processing their WCO to biodiesel. It is very costly to transfer WCO to the plant and processed or buy it in the form of biodiesel. The company sells the WCO because the WCO is reprocess and sold as reusable cooking oil. The current problem is that reprocessing WCO to biodiesel is costly and complicated. There is a need to simplify and encourage the food producing factory to do this process at their plant. A mini plant in the locality of the factory was built to save the cost. This project is purposed to fulfill the main objectives of reducing the environmental problem of WCO and also producing biodiesel for the usage of the factory. Diesel engine 5% biodiesel that meets the ASTM D975 standard and 95% petroleum diesel that meet the ASTM

D6751 standard to avoid damage and problems caused by external factors or elements they do not produce or control, such as the type of fuel or additives used in the engine.

1.3 Objectives of Project

The main objectives of this study are:

i. To study the operation of the plant with WCO as input with various molar ratio.
ii. To compare the properties of the biodiesel relative to ASTM D7461 and EN 14214.
iii. To evaluate and make comparison between plants blended biodiesel B5 and petroleum diesel B5 with ASTM D975.
iv. Evaluate economic to produce of biodiesel.

1.4 Scope of Project

This research focus on study of molar ratio of alcohol and characteristics of biodiesel produced from WCO at Azhar Food factory plant in Batu Pahat. The quality of the biodiesel produced through this research will determine the success of this project.

i. The input to the process is waste cooking oil from Azhar Food factory.
ii. The WCO tested were reacted with various molar ratio of alcohol.
iii. The testing of properties of biodiesel will be tested in the laboratories of UTHM.
iv. Analysis of properties based on density, kinematic viscosity, water content, acid value and flash point will be carried out.
v. The testing of properties of biodiesel will be tested in the laboratories of UTHM
vi. Properties blending of 95% petroleum diesel and 5% of biodiesel

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction palm oil in Malaysia

In early 1980s, the commercial interest in biodiesel started as the largest producer and exporter of palm oil. Its potential in the biodiesel industry was realized by the Malaysian government, thus making it a pioneer in that field (Lim and Teong, 2010). In 1982, Laboratory research on palm biodiesel, spearheaded by the Malaysian Palm Oil Board (MPOB) and funded by a research and development levy on the palm oil industry (Lopez and Laan, 2008) and in 2010, a pilot plant for producing palm biodiesel was constructed in collaboration with Petronas (Petroliam Nasional Bhd) (Lim and Teong, 2010).

The government adopted the National Biofuel Policy in 2006, to further promote the production and consumption of biodiesels. In support of the policy, the government announced a pledge to set aside 6 million tonnes of CPO for biodiesel production (MIDA, 2006). In August the same year, Malaysia's first commercial-scale biodiesel plant began operations in Pasir Gudang, Johor (Abdullah *et al.*, 2009). During August–December 2006, 55 000 tonnes of biodiesel was produced in Malaysia. This increased to almost 130 000 tonnes in 2007 (MPOB 2008). By the end of September 2007, the government had approved 92 licences for individual biodiesel projects with a combined production capacity of 10.2 million tonnes (Lopex and Laan, 2008). However, many of these proposed projects have been delayed or cancelled were increasing palm oil prices and decreases in fossil fuel prices (Abdullah *et al.*, 2009).

The Minister of Plantation Industries and Commodities announced that only 14 biodiesel plants were in production with a combined installed capacity of 1.68 million tonnes in October 2008. (Zuraimi, 2008), realising only 16% of the total production volume that was licenced in 2007. In 2009, exports of Malaysian biodiesel increased by 24.9% to 230 000 tonnes, from 180000 tonnes in 2008 (Basri, 2010). According to the MPOB, MYR605.8 million in 2009 the export revenue generated from biodiesel. Nevertheless, less than 1% of palm oil produced in Malaysia is used in biodiesel production (SOPPOA, 2009).

In January 2010, the Malaysian government had originally deadline to sell B5 biodiesel at all petrol stations nationwide. Due to high palm oil prices and consequently the large government subsidy needed (estimated at MYR250 million per year) to blend and distribute the biodiesel, the government also considered reducing the B5 blend to a B3 blend (Star Biz, 2009; Ooi, 2010). Understandably, the proposal was heavily criticised by biodiesel producers as a B3 mandate would mean using only 300 000 tonnes of biodiesel, too little to make production economically viable (Hanim, 2009b; Lim, 2010). Consequently, the government reverted to the original mandate of using the B5 blend. Implementation was delayed to June 2011 and limited to the central region comprising Kuala Lumpur, Melaka, Negeri Sembilan, North Johor, Putrajaya, Selangor, Southern Perak and West Pahang (Dompok, 2010). Malaysia now has 25 biodiesel plants that operating in peninsula Malaysia.

2.2 Feedstock waste cooking oil

In 2008, Malaysia produced 17.7 million tonnes of palm oil on 4.5 million hectares and was the second largest producer of palm oil. Biodiesel can be produced from any material that contains fatty acids that linked to other molecules or present as free fatty acids. Thus various vegetable fats and oils, waste greases, animal fats and waste oil can be used as feedstock for biodiesel production. The choice of feedstock is based on local availability, cost, and government support and fuel performance.

In the fast food business, a single branch which serves fried food such as fried chicken, French fries and burgers can produce as much as 15 litres of WCO per day. Consider that there are hundreds of these outlets in Malaysia, the total amount generated can reach several thousand litres per day. Properties of degraded used cooking oil after it gets into sewage system are conductive to corrosion of metal and it also affects to installations in waste water treatment plants (Darwin *et al.*, 2010). WCO is one of alternative source for biodiesel production. Biodiesel from WCO can reduce the cost of biodiesel production since the feedstock costs approximately 70-95% of the overall cost of biodiesel production (Zhang *et al.*, 2003). Although, biodiesel cannot entirely replace petroleum-based on diesel fuel, there are at least five reasons that justify its development such as:

i. It provides a market for excess production of vegetable oils and animal fats.
ii. It decreases even will not eliminate and the country's dependence on imported petroleum.
iii. Biodiesel did not contribute to the global warming due to its closed carbon cycle. A life cycle analysis of biodiesel shows that overall CO_2 emissions were reduced by 78% compared with petroleum-based diesel fuel (Barnwal, 2004).
iv. The exhaust emissions of CO, HC, and particulate emissions from biodiesel are lower than regular diesel fuel. Unfortunately, most emissions tests have shown a slight increase in nitrogen oxides (NO_x). When added to regular diesel fuel in an amount equal to 1–2%, it can convert to the fuel with poor lubricating properties such as modern ultra-low-sulphur diesel fuel.

2.3 Biodiesel

Biodiesel is an alternative fuel for diesel engine which is produced from straight vegetable oil, animal oil and fats, tallow as well as waste cooking oil reacted with an alcohol such as methanol. The reaction requires a catalyst that usually a strong base such as sodium hydroxide or potassium hydroxide and produces new chemical compounds known as methyl esters or biodiesel. Most biodiesel produced from waste vegetable oil sources such as from restaurants, streets shops, and industrial food producers. However, straight oil from the agricultural industry represents the greatest potential source and it is not being produced commercially simply because the raw oil is too expensive also most are food source. After the cost of converting it to biodiesel has been added on it is simply too expensive to compete with diesel. Waste vegetable oil can often be sourced for free or sourced already commercial for a small price and free. The waste oil must be treated before conversion to biodiesel in order to remove impurities. The result of biodiesel produced from waste vegetable oil can compete with diesel.

A primary feedstock from vegetable oil or animal fat is generally considered to be renewable. Since the carbon in the oil or fat originated mostly from carbon dioxide in the air, biodiesel is considered less contribute to global warming than fossil fuels. Diesel engines operated on biodiesel have lower emissions of carbon monoxide, particulate matter, unburned hydrocarbons and air toxics as compared when operated on petroleum-based diesel fuel (Gerpen, 2004).These requirements are listed in Table 2.1 for diesel and biodiesel defined by the specification. Specification D975 and D6751 also contains the standard test methods used to measure the values of the properties. The D975 and D6751 specification contains the minimum mandatory requirements needed to guarantee acceptable performance for the majority of users. In addition, this specification recognizes some requirements established by the EPA to reduce emissions. (Chevron Corporation, 2007)

Biodiesel as defined by the American Society for Testing and Materials (ASTM) and European Standard (EN), is a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats. Due to the great molecular similarities between biodiesel and diesel, this alternative fuel has a chance of fulfilling the technical requirements of diesel fuel it is a domestic, renewable liquid fuel, clean-burning that can be used in compression ignition engines instead of diesel with little or no modifications. The dominant stage of biodiesel production process which namely by transesterification. It typically involving the reaction of an alkyl-alcohol with a long chain ester linkage in the presence of a catalyst to yield mono-alkyl esters (biodiesel) and glycerol (Saka and Kusdiana, 2001).

Table 2.1: ASTM D6751 and D975 Standards of maximum allowed quantities in diesel and biodiesel (Lotero and Kiss, 2005)

Property	**Diesel**	**Biodiesel**
Standard	ASTM D975	ASTM D6751
Composition	HC[a] (C10-C21)	FAME[b] (C12-C22)
Kinematics viscosity (mm^2/s) at 40°C	1.9-4.1	1.9-6.0
Boiling point (°C)	188-343	182-338
Flash point (°C)	60-80	100-170

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

It can be concluded that first objective of producing biodiesel using that small plant has also been achieved using the WCO of the factories with various molar ratio of alcohol. The biodiesel produced are as planed and good for use for factory.

The second objective of the research is the comparison biodiesel properties against ASTM D6751 were also carried out. A total of 9 samples were successfully produced biodiesel fuel, which used three different ratios which is at 6:1, 9:1 and 12:1 with a reaction time of 120 minutes. The reaction temperature was set at around 65°C and found that of the resultant biodiesel satisfy with kinematic viscosity, acid value, water content, FFA and flash point temperature. This has proven that the designed plant and the process using WCO for this project has been successful. The biodiesel produced by the plant value has fulfilled the EN14103 standard were molar ratio of alcohol 6:1 is 97.8% meanwhile molar ratio 9:1 value is 97.2 % and molar ratio 12:1 is 95.7% has been achieved. This showed that the plant has successfully produced biodiesel from WCO at Azhar Food and satisfy the en14103 standard. The best yield of methyl ester from waste cooking oil is 6:1 molar ratio should be used for conversion to biodiesel.

For third objective, the comparison of 5 percent of biodiesel from the plant with commercial petroleum B5 biodiesel, the results are similar and this further proved the viability of the biodiesel plant. The whole project is completed and successful.

5.2 Recommendations

In this study there are a few suggestions that can be considered as a guide for future studies.

1. In future studies, ultrasonic are use for processing biodiesel for save energy consumption and cost compare to mechanical stirring that processes take 3 day to produce biodiesel.
2. The heterogeneous solid catalyst in next studies will be explored using mechanical stirring.
3. Studies with different catalyst concentration, temperature, speed of agitator using mechanical stirring to satisfy a mini plant.
4. The results of this study can also be used to evaluate the performance of biodiesel based on waste cooking oil (WCO). Biodiesel from waste cooking oil may be tested on the vehicle engine to find out whether it is suitable for use in a vehicle.
5. The plant can be operated more extensively to evaluate the performance over a long period time.
6. Upgrade the equipment such as release valve, automatic cut off pressure, pump, and piping to make it more efficient.

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